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# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

# Manufacture of Aqueous Solutions of Chlorous Acid

We, CHLORATOR G.M.B.H., of Grötzingen near Karlsruhe, Baden, Germany, a Body Corporate organised under the Laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the manufacture of aqueous solutions of chlorous acid and in particular is directed to, and has for its principal object, a process for the production of stable aqueous solutions of that acid.

Salts of chlorous acid such as sodium chlorite, $NaClO_2$, are known compounds, but it has hitherto not been possible to manufacture the free chlorous acid, $HClO_2$, and to investigate its properties. This is probably due to the fact that the usual way of preparing acids by decomposition of their salts by reaction with stronger acids leads in the case of salts of chlorous acid to the formation of chlorine and water besides the salt of the acid used for the decomposition.

It has been found by experiments that aqueous solutions of chlorous acid may be obtained without any or any substantial decomposition of said acid in a simple and inexpensive manner by reaction of aqueous solutions of salts of chlorous acid with hydrogen-ion-exchangers and that the aqueous solutions of chlorous acid thus obtained are stable and may be used as oxidising agents for example for the purification and sterilisation of water or sewage or as bleaching agents.

The invention is based upon these experiments and provides a process for the manufacture of an aqueous solution of chlorous acid in which an aqueous solution of a salt of chlorous acid is reacted with an ion-exchanger containing hydrogen as exchangeable ion.

The most suitable water-soluble salts of chlorous acid for use in the process according to the invention are the alkali metal chlorites, especially sodium chlorite, and it is preferred to use the pure salts, rather than technical products, in order to obtain high yields of chlorous acid in the form of aqueous solutions thereof. Any ion-exchanger containing hydrogen as exchangeable ion and stable against oxidising agents may be used. Preferred ion-exchangers for use in the process are the synthetic ion-exchanger resins, such as the products known under the Trade Marks Lewatit S 100 and Amberlite IR 120 (Lewatit and Amberlite being Registered Trade Marks used in respect of resinous $SO_3H$-substituted polystyrene resins), in their hydrogen form, but synthetic or natural zeolites in their hydrogen form may also be used as ion-exchangers. The aqueous solutions of salts of chlorous acid used in the process according to the invention are preferably dilute solutions which do not contain more than 1.5% and preferably contain 0.8-1% by weight of these salts.

The preferred embodiment of the process according to the invention consists in passing an aqueous solution of a salt of chlorous acid having a temperature of not more than 30°C. through a layer of the ion exchanger with a rate of flow of not more than 1 ml solution per ml ion-exchanger per minute.

By the reaction between the salt of chlorous acid and the hydrogen-ion-exchanger in the process according to the invention, chlorous acid ($HClO_2$) is formed and the ion-exchanger is converted into an ion-exchanger which contains the cation of the salt used as exchangeable ion. This ion-exchanger

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may easily be reconverted into an ion-exchanger containing hydrogen as exchangeable ion by treatment with a dilute acid, such as aqueous hydrochloric acid, and the hydrogen-ion-exchanger thus obtained may be used again in the process according to the invention.

The following is an illustrative example in accordance with the invention.

EXAMPLE

A cylindrical vessel, having an inside diameter of 80 mm. and a length of 30 cm., is charged with one litre of a resinous ion-exchanger such as Lewatit S 100 as available commercially. The ion-exchanger is first treated with a 10 per cent. aqueous solution of hydrochloric acid so as to obtain the hydrogen-form of the exchanger and is then washed with desalted or distilled water in order to remove any hydrochloric acid and/or chloride present. 9 Litres of a 1 per cent. aqueous sodium chlorite solution, having a temperature below 30°C., are passed in about one hour through the column of the exchanger in the vessel whereby a stable aqueous solution of chlorous acid is obtained. The ion-exchanger, which contains sodium as exchangeable ion, is reconverted into an hydrogen-ion-exchanger by the above described treatment with a 10 per cent. aqueous solution of hydrochloric acid and thereafter again used for the manufacture of further amounts of aqueous solution of chlorous acid.

By diluting the aqueous solution of chlorous acid thus obtained with a large amount of water the chlorous acid is decomposed with formation of ozone, hypochlorous acid and hydrochloric acid.

The use of the aqueous solution of chlorous acid obtained as oxidising agent is based upon this decomposition and may be illustrated by the following example:

The aqueous solution of chlorous acid obtained which contains about 1 per cent. of this acid is diluted by addition of about 4500 litres of salt-free water and the bleaching liquid thus obtained is used for the treatment of cellulose fibre. The bleaching process is completed in about 10 minutes.

The rate of decomposition of the chlorous acid by dilution of the aqueous solution of chlorous acid obtained with water may be increased by the presence of an acid substance and decreased by the presence of an alkaline substance and the addition of one of these substances may be used for regulating the rate of decomposition.

Our copending Application No. 16267/56 (Serial No. 791,681) of even date describes and claims *inter alia* a method of purifying water or sewage in which an aqueous solution containing a salt of chlorous acid is converted by reaction with an ion-exchanger containing hydrogen as exchangeable ion into an aqueous solution containing free chlorous acid and the latter is added to the water or sewage to be treated.

What we claim is:—

1. A process for the manufacture of an aqueous solution of chlorous acid in which an aqueous solution of a salt of chlorous acid is reacted with an ion-exchanger containing hydrogen as exchangeable ion.

2. A process as claimed in Claim 1, wherein an aqueous solution of an alkali metal chlorite is employed.

3. A process as claimed in Claim 2, wherein an aqueous solution of sodium chlorite is employed.

4. A process as claimed in any of the preceding claims, wherein an aqueous solution of a pure chlorous acid salt is employed.

5. A process as claimed in any of the preceding claims, wherein an aqueous solution of not more than 1.5% by weight of a salt of chlorous acid is employed.

6. A process as claimed in Claim 5, wherein an aqueous solution of 0.8-1% by weight of a salt of chlorous acid is employed.

7. A process as claimed in any of the preceding claims in which an aqueous solution of salt of chlorous acid having a temperature of not more than 30°C. is passed through a layer of the ion-exchanger with a rate of flow of not more than 1 ml solution per ml ion-exchanger per minute.

8. A process as claimed in any of the preceding claims, wherein a synthetic ion-exchanger resin is employed.

9. Aqueous solutions of chlorous acid whenever produced by the process claimed in any of the preceding claims.

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